

Comments

Comment on “Spin-Coated Periodic Mesoporous Organosilica Thin Films with Molecular-Scale Order within the Organosilica Wall”

The recent publication in this journal of work by Wu et al.¹ reports preparation of mesoporous benzene-silica thin films with molecular-scale periodicity. However, we think the experimental results do not properly support the conclusion that the pore walls consist of the periodically arranged benzene-bridged bissilanetriols (Ph-triols) but indicate that the obtained film is a blend of a mesoporous thin film with amorphous pore walls along with a small fraction of nonporous Ph-triol particles having molecular-scale periodicity. First of all, few T⁰ (R-Si(OH)₃; triol) species are detected in the ²⁹Si MAS NMR spectrum (Figure 3), indicating the triol groups are in minor extent, contradicting the authors' conclusion. Additionally, such a low degree of condensation (mainly T² species) is typical for organosilica materials prepared by sol-gel synthesis under acidic conditions² and

not a favorable reason to elongate *d*-spacing of the molecular-scale periodicity to 10.1 Å. Second, the XRD peaks at 10° < 2θ < 50° due to periodic arrangement of the Ph-triols (Figure 1b,c) are very sharp despite the fact that the molecular-scale periodicity in the thin pore walls (e.g., a few nanometers) usually results in broadening of the XRD peaks,³ suggesting that the periodic arrangement of the Ph-triols is not formed in the pore walls. Finally, the lattice fringes appear to run independently of the pore channel patterns and also change direction in the same channels⁴ in the TEM image (Figure 4b), which is not likely to occur in mesoporous materials with molecular-scale periodicity and may result from overlapping of two phases.

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(2) (a) Shea, K. J.; Loy, D. A.; Webster, O. *J. Am. Chem. Soc.* **1992**, *114*, 6700. (b) Fan, H. Y.; Lu, Y. F.; Stump, A.; Reed, S. T.; Baer, T.; Schunk, R.; Perez-Luna, V.; Lopez, G. P.; Brinker, C. J. *Nature* **2000**, *405*, 56.

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- (3) Inagaki, S.; Guan, S.; Ohsuna, T.; Terasaki, O. *Nature* **2002**, *416*, 304.
(4) The lattice fringes run from top-left to bottom-right in the upper part whereas they run from bottom-left to top-right in the lower parts in the same pore channels (see the left side of Figure 4b).